Preparation, Characterization and its Application for Energy Production

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Abstract

Ferro fluids are colloidal suspensions of magnetic particles which respond to external magnetic field enabling the possibility of changing the magnetic flux by moving an external magnet about it. In the present work Fe_3O_4 nano magnetic particles are first synthesized in lab using co-precipitation technique followed by ferrofluid preparation. Structural and magnetic properties are characterized by using XRD, TEM, FTIR and VSM. Their ability to produce a fluid phase of magnetic material is used to demonstrate the generation of energy using a simple energy harvester setup and this energy is demonstrated to produce useful work eg, glowing of a LED.

Keywords: Ferro-fluid, nano magnetic material, Co-precipitation

1. INTRODUCTION

Although non stable magnetic fluids were being prepared as early as, the first successful synthesis of magnetic fluids was reported by in 1965. The study of magnetic fluids has since then become an ever-growing area of multi disciplinary interest with chemists, physicists, biologists and engineers engaged in the synthesis and characterization and evolving technological applications for these wonder materials [1-3]. The ability of a ferro fluid to produce patterns of nano scale magnetic particles in liquid phase which can be monitored by an external magnetic field has made it a viable option for application in many technological areas. The most prominent being memory storage, drug delivery, energy production etc [4-6].

In the present work, the magnetic particles are prepared by co-precipitation technique. The ferromagnetic particles of fe_3O_4 are suspended in the carrier fluid and coated with a surfactant as shown in Fig. 1, to ensure they do not clump together. A simple experimental set up is designed of a vibratory energy harvester utilizing a ferrofluid sloshing in a cylindrical plastic tank. Mechanical vibrations change the orientational order of the magnetic dipoles creating a varying magnetic flux thus producing electric current as per Faraday's laws. The ferrofluid was successfully synthesised and the experimental results show generation of voltage drop of around 2V at vibratory frequencies as low as 4-5 Hz

2. EXPERIMENTS

2.1 Synthesis of Fe_3O_4 Nano particles and Ferro fluid

The co-precipitation method was used to synthesize ferrofluid. The process for the Fe_3O_4 nucleation from the salt solution occurs in accordance with the reaction:

$$2FeCl_3.6H_2O + FeCl_2.4H_2O + 8NH_4OH \longrightarrow Fe_3O_4 + 8NH_4Cl + 20H_2O$$

A solution of $FeCl_3$.$6H_2O$ (0.5 M i.e 10.35mL in 110mL water) and $FeCl_2.4H_2O$ (0.5 M i.e 6.85g in 60mL water) mixed in a molar ratio of 2:1 was prepared in contact with air. An ammonia aqueous solution (25%) of 15 ml was then quickly charged into the solution with vigorous stirring using a mechanical stirrer, followed by adding more ammonia aqueous solution into the mixture (app. 10mL) while stirring. The solution was stirred for about one hour. A black precipitate was obtained. Oleic acid (5% v/v) about 30mL was added slowly and intensely stirred at 60 ^{0}C for 30 min. The precipitate was separated using a magnet and washed with deionised water and methanol several times. The ferrofluid solution was centrifuged at low rpm for ten minutes .precipitate was dried at 70 ^{0}C for 3 hours and the appropriate amount (7% v/v) of solid phase was dispersed in kerosene oil.

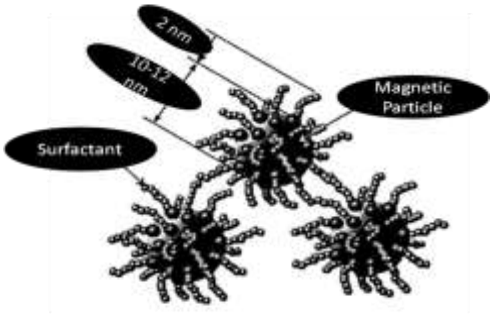

Fig. 1- *The ferromagnetic particles of fe_3O_4 are suspended in the carrier fluid and coated with a surfactant*

2.2 Characterization

The structural characterization of samples was carried out by the X-ray diffraction (XRD Rigaku Miniflex II, step size = 0.02) technique using CuKα radiation (λ = 1.5406 Å). The average crystalline size was determined from the measured width of their diffraction curves using the Debye Scherrer's relation: $D = 0.9\lambda/\beta \cos\theta$, where λ is the wavelength (λ= 1.5406 Å) of the CuKα radiation and β is the full width half maximum (FWHM) in radians calculated using Gaussian fitting. Particle size of the nano Fe_3O_4 samples was evaluated by using TEM. FTIR spectra of sample was recorded in KBr medium in the wave number range 400–4000cm^{-1} using a PerkinElmer FTIR (spectrum BX).

Magnetic measurements were performed at room temperature by plotting M-H curve for the samples using vibrating sample magnetometer for all samples. The magnetic properties such as saturation magnetization (Ms), Coercivity (Hc) and remanence (Mr) were determined from M-H curves.

2.3 Application

A simple experimental setup was designed to show the ability of a changing magnetic flux produced by the ferro fluid to produce useful energy that can be made to do work eg, lighting of an LED. The setup is shown in Fig. 2. The vibratory energy harvester consists of a conducting copper coil wound around an insulating plastic container tank filled with the ferro fluid. Rare earth magnet fillets were put in the ferrofluid so that on starting the vibratory motion, a changing magnetic flux is produced. The two ends of the coil are connected first to a bridge rectifier supplemented with a capacitor of sufficiently high value to render the ac voltage produced to a non fluctuating dc voltage. The output of the capacitor filter is then connected to a measuring device like digital multimeter or LED.

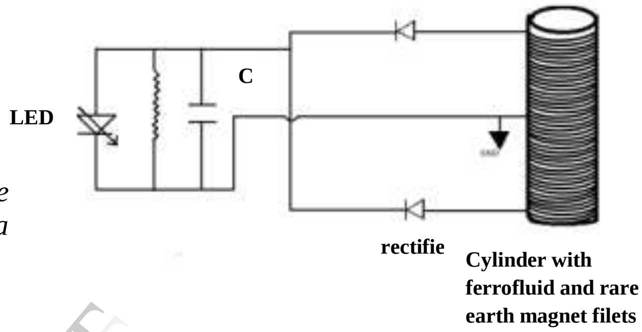

Fig. 2 – *Energy harvester setup*

3. RESULTS AND DISCUSSION

3.1 Structural Analysis

Fig. 3 exhibit the XRD patterns of nano Fe_3O_4 prepared by co-precipitation techniques. It is evident that the as-prepared powder of Fe_3O_4 is in single phases confirming all the peaks in the pattern matching well with JCPDS card [7]. The average crystallite size of Fe_3O_4 sample was found about 12nm calculated from Debye Scherrer's relation which is supported by the TEM analysis. Maximum numbers of the particles were found in the range of the 9-12 nm, as shown in the fig. 4. Distribution of the particles size and selected area electron diffraction pattern is shown in the in-set of fig. 4.

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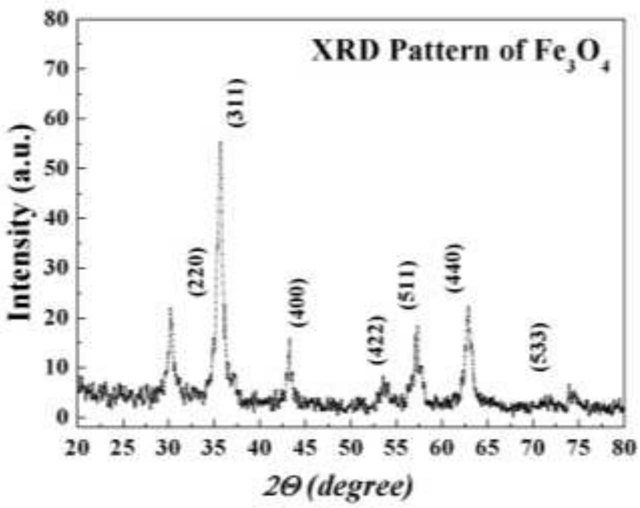

***Fig. 3** – XRD pattern of the Fe_3O_4nano-particles prepared by co-precipitation technique.*

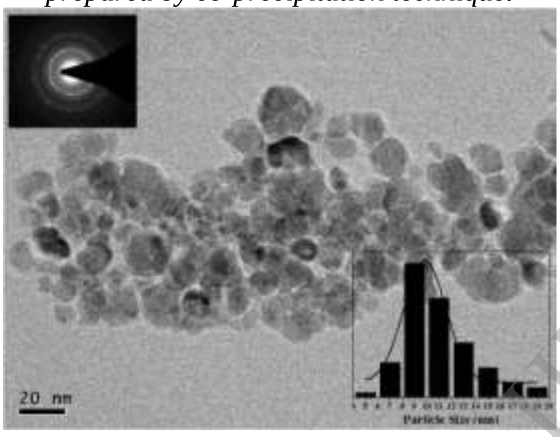

***Fig. 4.** TEM analysis of the Fe_3O_4nano-particles prepared by co-precipitation technique*

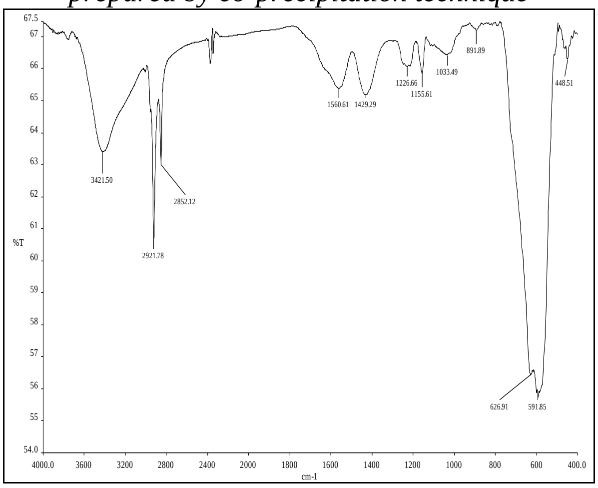

Fig. 5 – *FTIR spectra of the Fe_3O_4nano-particles prepared by co-precipitation technique*.

Fig. 5. shows the FTIR spectra of prepared nano Fe_3O_4 samples prepared by co-precipitation techniques. The presence of wave number bands in the range of (400-600 cm^{-1}) in the spectra, confirm the formation of Fe_3O_4 [8]. As prepared samples A, B and C showed the characteristic bands at about 1300 & 2900 cm^{-1} corresponding to the NO_3^- ion and O-H group respectively. FTIR curve for sample shows the formation of spinel structure.

3.2 Magnetic characterization

The hysteresis curves (M-H curves) for the Fe_3O_4 nano-particles prepared by Co-precipitation is shown in Fig. 5. Due to the applied magnetic field (*H*), the magnetic induction (*B*) could be measured according to Faraday's law, and the magnetization (*M*) was then obtained by the relationship:

$B = H + 4\pi M$

The applied magnetic field was increased until the saturation magnetization was achieved. Then the applied field was reduced to zero to measure the remanence (residual magnetization). When the applied field was further reversed, the coercivity (the applied field that reduces magnetization to zero) and the saturation magnetization in the reverse direction could be obtained. This increasing and decreasing applied field process was repeated five times to get the magnetization curve (magnetization (*M*) versus applied magnetic field (*H*)) and examine the magnetic properties of the Fe3O4 particles. In addition, the important parameters used to characterize the magnetic properties of solids, magnetic susceptibility (χ), and magnetic permeability (μ) were also calculated using the following equations:

$\chi = M/H$

$\mu = B/M$

Parameters such as saturation magnetization (Ms) and coercivity (Hc) were determined from the hysteresis curves. The saturation magnetization of sample is 48.8 emu/g at room temperature. The area within a M-H loop represents a magnetic energy loss; this energy loss is defined as heat that is capable of rising specimen temperature. It is observed that area of the M-H curve is small, and the loop is thin and narrow which is a specific criteria for soft magnetic material and required for the ferro fluids preparation. The magnetic response of the ferro fluid in the presence of the external magnetic is shown in the Fig. 7.

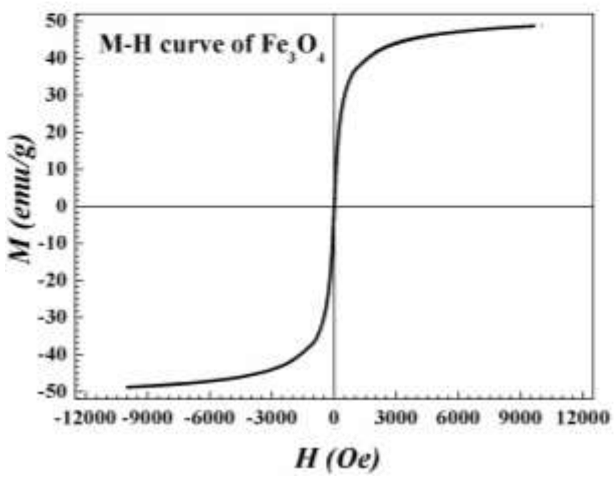

Fig. 6 - *M-H curve of the Fe_3O_4nano-particles prepared by co-precipitation technique.*

Fig. 7 - *Response of the ferrofluid in the presence of the external magnetic field.*

3.3 Energy Production

It was observed that vibrating the ferrofluid filled cylinder with a frequency of as low as 5-10 Hz produced sufficient current in the conducting wire to light up an LED or equivalently to generate a 2V drop across the wire ends.

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